NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 2224

MULTIPLE-FILM BACK-REFLECTION CAMERA FOR ATOMIC STRAIN STUDIES

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SUMMARY

A new back-reflection X-ray diffraction technique, which eliminates some of the principal limitations and reduces the remaining limitations imposed by conventional single-film back-reflection methods, was developed through use of a multiple-film camera containing four parallel films separated by known distances. Diffraction angles were calculated by determining the change in radius of the diffraction ring from film to film. In the analysis of a polycrystalline aggregate, the atomic spacing of a particular set of crystal planes in essentially one orientation is determined from the calculated diffraction angle. Diffuse diffraction patterns could be analyzed by the multiple-film technique with greater accuracy than could be obtained with conventional cameras. Calibration of the multiple-film camera with a gold powder standard for a set of planes having a reported atomic spacing of 0.91008 A yielded a possible accuracy of the atomic spacing of approximately $\pm 4 \times 10^{-5}$ A.

A multiple-film-technique analysis and a conventional-method analysis of the same X-ray strain data indicated that a more detailed analysis of atomic strain could be obtained from the multiple-film technique.

INTRODUCTION

The application of X-ray diffraction techniques to a nondestructive method of stress analysis was made as early as 1925 (reference 1); in 1930, back-reflection cameras were used to show that, under favorable conditions, accuracies obtained from X-ray strain measurements of the crystalline lattice were comparable with accuracies obtained from various types of strain gages (reference 2). The principles of X-ray strain measurement are described in reference 3, which shows how only the back-reflection technique can yield a reasonable strain accuracy.

Because the conventional experimental methods reduce the precision in determining interatomic spacing and restrict the analysis to those materials yielding reasonably sharp diffraction patterns, a new back-reflection camera and technique have been devised at the NACA Lewis

laboratory. The multiple-film camera and the technique, which permit precision determination of the lattice spacing for the diffuse as well as the sharp diffraction patterns, were used in a two-exposure stress study of SAE X4130 steel and the results compared with those obtained from conventional analysis.

APPARATUS AND TECHNIQUE

The back-reflection geometry involved when a beam of parallel X-rays of a single wavelength strikes a polycrystalline specimen is presented in figure 1. The beam selects only those crystals that are oriented to satisfy the Bragg reflection and the resulting diffraction from two different orientations of crystal planes is as shown in figure 1. Any reference plane containing the incident beam will also contain diffracted beams from other crystals properly oriented so that the diffraction is exhibited as a cone in three dimensions or as a ring on the back-reflection camera film that intercepts the cone of rays. If the specimen is completely homogeneous and without internal or external stresses, the diffraction ring on the back-reflection film is a perfect circle. If, however, internal stresses are present or if externally applied loads result in a strain distribution among the crystals, all the diffracting planes no longer have the same diffraction angle and the diffraction ring is no longer a circle. Precision measurement of ring radius and the film-to-specimen distance is needed to calculate the diffraction angle by conventional methods. The difference between this angle and the diffraction angle at zero strain indicates the state of strain of the diffracting crystals oriented to give that particular portion of the diffraction ring.

The geometric principle governing the multiple-film back-reflection method is shown in figure 2 in which two (or more) flat films are mounted parallel to one another and separated by known distances. Simultaneous exposure of all films to the diverging cone of rays yields successively larger diffraction rings; the analysis of any two films determines the diffraction angle. A multiple-film camera, designed to operate on this principle, is shown in figure 3. Four studs on the back of the camera mounting plate are used to mount the camera in front of the X-ray tube. The small screws at the back and on the sides of the mounting plate provide adjustment for the six segmeted bronze bearings that support the rear and outer bearing surface of film plate IV. A bronze bearing ring, fastened to the front of the mounting plate, restricts the front bearing surface of film plate IV. A cellulose dust shield is placed over this bearing ring.

Film plate IV has a central hole for insertion of the removable front and rear collimating tubes; this hole is concentric with the center post and the outer bearing surface. An annular brass gear mounted on the periphery allows the entire camera to be oscillated by a small electric motor within the limits defined by the screw stops on the gear surface. The film cover plate and film plates I to III, which are segmented to allow radiation to the fourth film, are positioned by dowels; all plates are attached by screws to film plate IV. A brass ring of special contour mounted on the film cover plate serves as a light trap in conjunction with a mating aluminum plate in the stationary light shield. The light shield, which clamps to the camera housing with three spring clips, serves the dual purpose of restricting the portion of the diffraction ring to be analyzed as well as keeping normal light from the film. The 30° opening in the stationary light shield is covered with black paper. The sector guard, mounted above the light shield on the center post of film plate IV, permits two exposures to be taken with each film set.

The film scriber rotates on the center post and the film plate surface, and the needle is adjusted by a small thumb screw. All critical parts of the camera and scriber were machined within a tolerance of ±0.0002 inch. A precision check showed that the diameter of the scribe circles could be measured within an average deviation from the mean of 0.003 millimeter, whereas the maximum deviation encountered was 0.007 millimeter.

EXPERIMENTAL PROCEDURE

Film for the camera is cut on special templates to the shapes shown in figure 4 in order to allow the diffraction pattern to reach each film without interference. Films are cut slightly oversize to prevent tilting of the plates by irregular film edges. In the loading procedure, a film is set in place, the plate above it is screwed into position, and an arc of known radius is scribed on the film in the area where it will be exposed. The other films are placed and scribed similarly, care being taken not to tilt the plates when tightening them (fig. 5). The stationary shield is snapped into place and then the sector guard is located to expose one-half of the film.

The camera is placed on the tube mount and alined to irradiate the desired portion of the specimen. An X-ray generator with portable tube provides the source of radiation. The camera mounting (fig. 6) is equipped with a drive motor and switch for camera oscillation. The specimen table pivots about a fixed post to allow control of beam-to-specimen angle. Loading is effected by a cantilever arrangement with hanging weights.

After one exposure is completed (approximately 2 hr), the sector guard is reset to expose the remaining film and the oscillation limits are accordingly changed. The camera is rotated 180° for this second exposure so that adjacent exposures on film I indicate opposite sides of the diffraction pattern. This procedure enables a correction to be made for plate tilting or beam misalinement. Let A_L and A_R indicate the atomic spacings as determined from the first exposure (left and right side of diffraction ring, respectively) and B_L and B_R indicate spacings determined from the second exposure (fig. 4). For diffraction from a standard powder, the condition $A_L = A_R = B_L = B_R$ indicates a proper beam alinement and plate positioning. However, if $B_L = A_R$, $A_L = B_R$, but $A_L \neq A_R$, the film plates are probably tilted; and if $A_L = B_L$, $A_R = B_R$, but $A_L \neq A_R$, a misalinement of the X-ray beam, which can consist of beam angularity and beam center deviation, is indicated.

The stationary shield permits only a portion of the diffraction pattern to be photographed, and each layer of film rotates completely through the irradiated area. Any radial sector on each film receives the same radiation as another sector on it or on any other film. In this manner, beam irregularity and variation in absorption of the black-paper light shield do not cause any irregularity in the pattern on the film. The only irregularity in the final pattern is due to film response and may be averaged by analysis along several radii.

When the camera is unloaded, the exposure number is scratched on the film. Film I is developed for the normal development time and films II to IV are over-developed for successively longer periods, respectively, so that the resulting density of all films is approximately equal. After processing and washing, films are spun dried to remove any water marks and then hung horizontally until completely dry. The films are then mounted in a special holding plate on a microphotometer where their density fluctuation is reproduced and magnified. A reference circle of 88.877 ±0.005 millimeters diameter on the holding plate allows an accurate shrinkage check in conjunction with the scribe circle on the film. Radius measurements are possible on the film because of the camera concentricity and the accuracy of the scribe circle.

CAMERA CALIBRATION

Because the diffraction radii on any two of the four diffraction films can define the diffraction angle, six values of the diffraction angle are possible. The average of these values is used to determine the atomic spacing. The consistency of these six readings, determined from the average percentage of deviation of the tangent of the diffraction

angle from the mean value, is an indication of the reproducibility of any single d-value in a given setup. For most of the data, this average deviation of the tangent was less than ±0.2 percent, which represents a difference in the spacing of the 310 planes in steel of about $\pm 4 \times 10^{-5}$ A.

Several calibration exposures were made on a gold-powder standard for which the atomic spacing of the 420 planes was 0.91008 ±0.00001 A (reference 4). The preliminary calibration with a 0.060-inch (0.152 mm) collimating system and cobalt Kα radiation yielded d-values from diffraction-ring radius measurements as follows:

	Exposure A (A)	Exposure B (A)
Left side of ring	A_L = 0.91013	B_L = 0.91009
Right side of ring	A_R = 0.91015	B_R = 0.91018
Ring diameter/2	.91014	.91014

Because $A_L = B_R \doteq B_L = B_R$ within the expected reproducibility of $\pm 4 \times 10^{-5}$ A, no misalinement of the X-ray beam or tilting of the film plates can be detected.

A sharper diffraction pattern was obtained from the same standard when 0.040-inch (0.102 mm) collimating pinholes were used and the cobalt X-ray tube was replaced with a new one. The following data were obtained:

	Exposure A (A)	Exposure B (A)
Left side of ring	A_L = 0.91022	B_L = 0.91022
Right side of ring	A_R = 0.90990	B_R = 0.90989
Ring diameter/2	.91006	.91005
Average d-value (corrected for misalinement)	.91007	.91006

Because $A_L = B_L$, $A_R = B_R$, but $A_L \neq A_R$, beam misalinement is present. The actual magnitude of the misalinement in only several hundredths of a degree. It is difficult to remove beam misalinement entirely in a portable-tube setup; correcting the misalinement can be achieved with the described diffraction method because it employs a precision radius measurement.

The possible accuracy is indicated by comparing the data obtained with the two collimators and the reference for a gold-powder standard. The maximum deviation from the mean of these three values is 4.3×10^{-5} A, which is in the same order of magnitude as the reproducibility in a given setup.

LIMITATIONS OF CONVENTIONAL BACK-REFLECTION TECHNIQUES

With a conventional single-film back-reflection camera, some means must be employed to determine film-to-specimen distance. Some investigators (references 3, 5, and 6) employ a calibrating substance (generally an annealed powder) of known atomic spacing that yields another diffraction ring on the back-reflection film. When the diffraction angle of the calibrating substance is known, the film-to-specimen distance can be calculated. A simple proportion between the radius of the unknown ring and the standard ring is sufficient to determine the unknown diffraction angle. Certain inaccuracies, however, are inherent in this method. If the standard powder is applied in the form of a paste to the specimen surface, extreme care must be exercised to maintain an equitable balance between the diffraction from the powder and from the specimen. The standard powder increases the general background level, which prohibits use of this method for specimens that do not yield high-contrast diffraction patterns. An alternative of this method is to apply the standard before or after exposure of the specimen by means of tape, but extreme care must be taken not to move the specimen during application. Also, the accuracy of placement of the standard within the required tolerances is questionable. Regardless of how the powder is applied, corrections are a necessity because the calibration ring actually indicates the distance to the effective surface of the calibrating substance, whereas the diffraction from the specimen indicates conditions existing perhaps 0.001 inch below the specimen surface. For exposures where the incident beam is perpendicular to the specimen surface, the correction would be the same for opposite sides of the diffraction ring; but for inclined exposures, a different correction would be necessary for each side of the ring. Few investigators have made measurements of diffraction radius and are content with assuming that measurement of one-half of the ring diameter is indicative of the average behavior of the two sets of crystal orientations being studied.

The application of powders to irregular surfaces also imposes limitations on the versatility of the method. Furthermore, analysis of such data is subject not only to error in the measurement of the specimen diffraction pattern but in the pattern of the calibrating substance as well.

Other investigators (references 7 and 8) have been dissatisfied with the use of calibrating substances and have devised special gages and fixtures for measuring film-to-specimen distance. A direct-acting contact gage should be avoided because contact at the point of X-ray impingement on the specimen surface could mar the special surface finish necessary for diffraction study. Some type of feeler gage must therefore be used between the contact gage and the specimen surface. This requirement limits the precision to the accuracy with which the feeler gage can be placed between the contact gage and the specimen. A precision dial indicator may be used as the contact gage and can give sufficient precision for measurements taken with the X-ray beam perpendicular to the specimen surface. For inclined exposures, however, the side play of the dial indicator as well as the rounded tip of the instrument must be considered. With this method, it is believed that precision within several ten thousandths of an inch is highly improbable. Realizing this, some investigators (for example, reference 7) attempted to measure the film-to-specimen distance along some other line parallel to the beam line; this method necessitates the use of special jigs and fixtures and places considerable limitation on the size and shape of the specimen being studied. Furthermore, with all mechanical distance gages the assumption is made that the beam is perfectly collimated. Any deviation in the effective beam from the center line of the collimator can cause considerable error in the interpretation of the inclined exposures.

A further objection to all these methods of film-to-specimen distance determination is that the setup must be so rigid that no movement occurs between film and specimen during exposure and after measurements have been made.

One of the outstanding limitations of the conventional back-reflection technique, which restricts both choice and preparation of specimen material, is that resonably sharp diffraction lines are necessary. A stress accuracy of ±3000 pounds per square inch in steel is considered very satisfactory (references 4 and 6). Unfortunately, material of sufficient strength for use in aircraft-engine components usually yields a low-contrast, diffuse diffraction pattern. Deep etching can improve the diffraction pattern from such a material but may also affect the lattice parameter (reference 3) or, in the case of a specimen under load, result in surface strain indications far less than the actual strain beneath the surface.

Analysis of the diffraction pattern to find the center of the peak presents several problems. For diffuse lines, the measurement of the center of the peak width at half-maximum intensity can result in an error in the determination of the α_1 peak as large as one-third the $\alpha_1\alpha_2$ peak separation (reference 9). Added to this error is one characteristic of X-ray film that is a considerable source of difficulty - the irregular

response of the relatively large-grained high-speed film. The microphotometer trace of a diffuse diffraction pattern (fig. 7) shows this irregularity and its relation to the peak height. Inasmuch as conventional analysis would not locate the peak position on the film (fig. 7) within ±0.01 millimeter, which represents the desired strain accuracy of $\pm 4 \times 10^{-5}$ A, new methods of analysis had to be devised. A strain accuracy of $\pm 4 \times 10^{-5}$ A represents ±2100 pounds per square inch in steel for the two-exposure stress method.

In the interest of short exposures, large beam sizes are employed in X-ray strain studies. An appreciable beam spread is expected, but, in addition, an angularity of the effective beam as well as an uneven intensity distribution across the beam may exist. Unless extensive calibrations are made that definitely establish the magnitude of these two components, errors in the determination of the diffraction angle will result from methods employing calibrating substances as well as those employing mechanical distance gages.

ADVANTAGES OF MULTIPLE-FILM TECHNIQUE

The advantages of the multiple-film technique over the conventional single-film methods are many. Complete elimination of accurately determining film-to-specimen distance also removes the limitations on the shape and size of the specimen to be studied. Furthermore, the film may be employed to best advantage by eliminating the extra diffraction pattern from the calibrating substance. Specimens having weak and diffuse patterns can be reliably analyzed by accurate location of the diffraction peak from the specimen alone. Absolute rigidity of the setup is less critical because any accidental movement that occurs during exposure will affect all films similarly.

The use of a stationary light shield is especially important for strain studies because the diffracted beam being photographed is limited to only those crystals in approximately the same stress field. The stationary shield also makes possible the analysis of the diffraction ring along any radius over the complete exposed sector, as previously described; therefore weaker and more diffuse patterns than can be analyzed by conventional methods may yield reliable results.

The multiple-film technique decreases the error associated with peak center determination. Inasmuch as the diffraction for any single wavelength can be assumed to be a parallel bundle of X-rays, the transposition from film to film of any representative portion of the peak should be the same. This similarity allows irregularity between the four films to be considered in the analysis. The technique employed in this investigation is to determine the center of area of the α_1 diffraction peak above some arbitrary base level established above the

α_2 peak. If diffuse diffraction patterns are encountered where the α_2 peak cannot be discerned and if the center of area of the peak above some arbitrary base level is used to indicate the transposition of the α_1 peak, an error in determining the diffraction angle will result because the peak being measured contains both α_1 and α_2 diffractions. The error may be reduced, however, by determining the transposition of some representative point in the area that is predominantly in the α_1 region.

Diffuse X-ray diffraction patterns have strongly limited conventional X-ray techniques as applied to strain studies. Difficulty of interpretation of diffuse patterns is to be expected. Because the diffuse pattern is indicative of a variance in the atomic spacings of a polycrystalline aggregate, no definite spacing of the atomic planes can be determined. The advantage of the multiple-film technique in this case lies in the reproducibility of the method because only relative values are required in strain analysis. Hence, any particular specimen yielding a diffuse pattern can be subjected to various loads and a reliable indication of its response can be obtained by investigating the behavior of this diffuse pattern with the multiple-film technique.

The difficulty of obtaining parallel collimation with large pinholes has been mentioned, and the effect of poor collimation in the multiple-film technique is small compared with the effect of similar collimation on single-film methods. Beam center deviation and beam angularity can be determined from a single exposure of a standard and corrections can be made for these factors in all other exposures with the same X-ray tube alinement.

APPLICATION OF MULTIPLE-FILM TECHNIQUE TO ATOMIC STRAIN STUDIES

A conventional two-exposure stress study was made on a cantilever bending specimen of SAE X4130 steel. The specimen was originally cut across the rolling direction of a hot-rolled plate and then carefully machined and ground so that each machining operation removed the cold-worked layer from the previous one. After quenching and tempering to a Rockwell C hardness of 30, the specimen was further treated by keeping it at a temperature slightly below the lower critical temperature and then cooling slowly, which resulted in a Rockwell C hardness of 23. The surface to be examined was metallurgically sanded and then lightly etched. This treatment produced a rather diffuse diffraction pattern (fig. 7) that was desirable to illustrate the use of the multiple-film camera at nonoptimum diffraction conditions. The specimen was then

loaded in bending as illustrated in figure 6 and at each stress condition two exposures were made with the multiple-film camera. One defining exposure was taken with the beam perpendicular to the specimen surface and the other with the beam at 45° to the surface.

The behavior of the 310 planes in SAE X4130 steel was obtained with cobalt Kα radiation and the 0.060-inch collimating system. Atomic spacings were calculated from the diameter measurements of the diffraction ring in each instance, and stresses were calculated from the conventional formula (reference 8):

$$\sigma_x = \left(\frac{d_\psi - d_\perp}{d_\perp}\right)\left(\frac{E}{1+\nu}\right)\left(\frac{1}{\sin^2\psi}\right)$$

where

σ_x X-ray stress (principal stress along intersection of specimen surface and a plane containing both incident X-ray beams)

d_ψ spacing for inclined exposure

$d_\perp$ spacing for perpendicular exposure

ψ incident angle for inclined exposure (45°)

E Young's modulus (3×10^7)

ν Poisson's ratio (0.28)

The results of these calculations are given in the following table:

	Applied stress, σ (lb/sq in.)	X-ray stress, σ_x (lb/sq in.)
Loading	0	-6,000
	30,000	29,000
	50,000	49,700
Unloading	30,000	30,600
	0	-4,700

Values in the preceding table seem to indicate that there was an initial compression in the specimen. The close agreement between calculated X-ray stresses and the applied stresses at 30,000 and 50,000 pounds per

square inch suggests that the component atomic strains at these points are also in accord with strains that can be calculated from the average elastic constants.

The component atomic spacings that were calculated from radius measurements of the same diffraction rings are presented in figure 8 where A_L and A_R are the d-values for the perpendicular exposure, left and right side of the diffraction ring, respectively, and B_L and B_R are the d-values for the corresponding inclined exposure. The dashed lines indicate the theoretical d-values as calculated for the different orientations from reference 7 as follows:

$$d = d_0 + \frac{\sigma}{E} d_0 \left[(1+\nu) \cos^2 \rho - \nu \right]$$

d atomic spacing at applied stress

d_0 atomic spacing at zero stress

σ applied stress

E Young's modulus (3×10^7)

ν Poisson's ratio (0.28)

ρ angle between perpendicular to 310 planes and direction of principal strain

The atomic planes of orientation A_R, B_L, and B_R shown in figure 8 behave in much the same fashion as predicted from calculations with macroscopic elastic constants. The planes of orientation A_L exhibit an increased strain acceptance.

A comparison of stress values given in the preceding table for a conventional two-exposure stress study with atomic spacing in figure 8 shows that: (1) Although X-ray stress values for 30,000 pounds per square inch are in very good agreement with applied stress, this agreement seems to be the result of compensating trends in the strain acceptance of the various planes. (2) The zero stress readings in the table indicate compression in the specimen, but, according to figure 8, only one set of planes (A_L) are contributing to this compression. Actually, this set of planes is indicating residual tension, whereas the other sets exhibit practically no residual stress.

Irregularities of the type just discussed might be present in any X-ray strain investigation but can be detected only by a back-reflection

method that allows accurate determination of diffraction ring radius. The multiple-film technique is one such method and can yield a more precise determination of atomic-strain behavior.

CONCLUDING REMARKS

Some of the principal limitations imposed by conventional back-reflection methods have been entirely removed and others considerably reduced by the multiple-film back-reflection technique. Patterns too diffuse for conventional analysis can be analyzed by the multiple-film technique to a precision comparable to that reported for sharp patterns with conventional methods. The problems, which then arise, concern the analysis of the behavior that is indicated by measurements thus made. The full significance of the behavior of a diffuse diffraction pattern from a material under residual stress or applied load has yet to be evaluated. The multiple-film technique is one method that can be employed in this evaluation.

Recently, more fundamental investigations of atomic stress-strain relations have been made that attest to the value of the X-ray diffraction technique for basic strain study. These studies were carried out with conventional techniques where diffraction diameters rather than diffraction radii were used. Such methods do not yield as complete an insight into the stress behavior of the crystallographic planes as would be obtained from radius measurements. The multiple-film back-reflection technique can yield a more exacting analysis of basic atomic strains.

Lewis Flight Propulsion Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, June 22, 1950.

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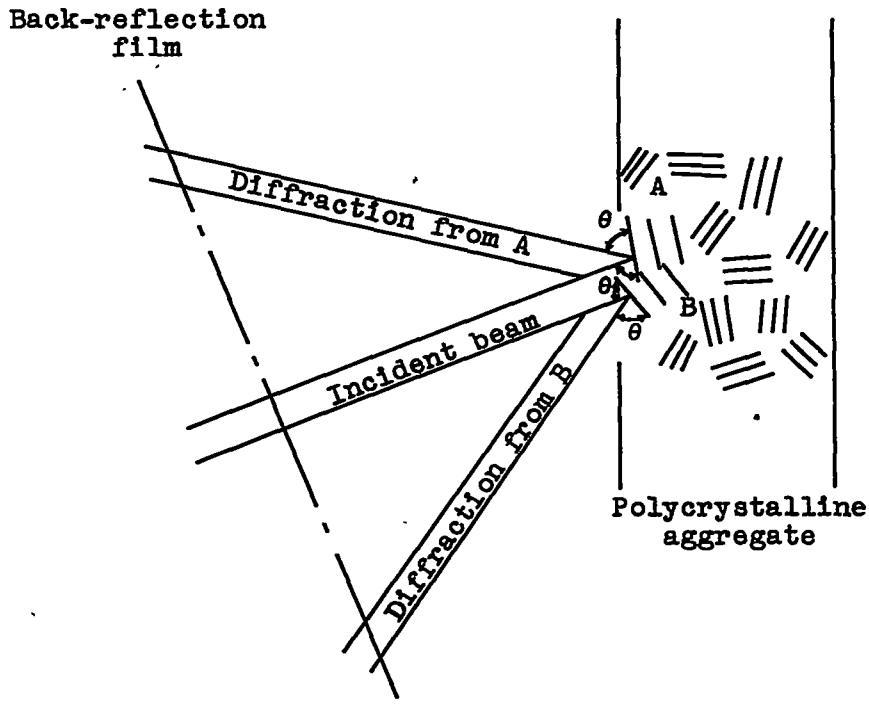

Figure 1. - Diffraction of monochromatic X-radiation from polycrystalline aggregate.

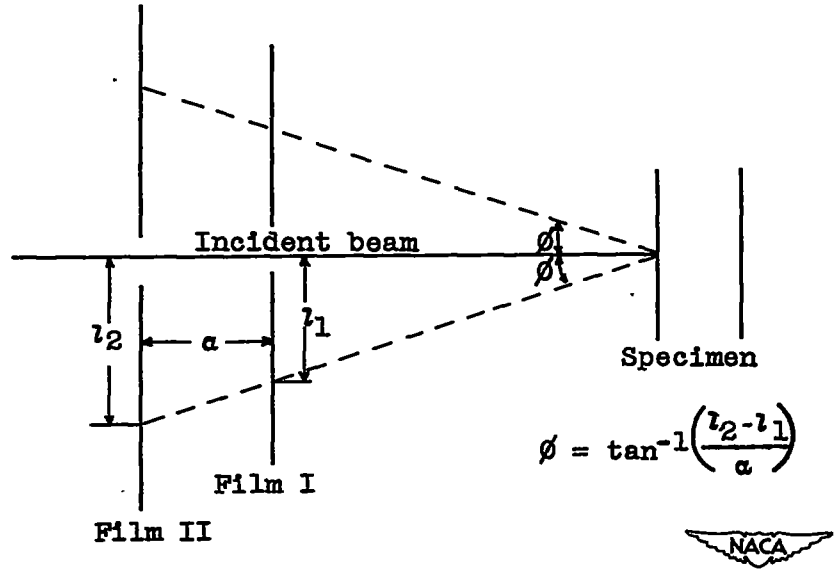

Figure 2. - Geometry of multiple-film camera.

Figure 5. - Components of multiple-film camera.

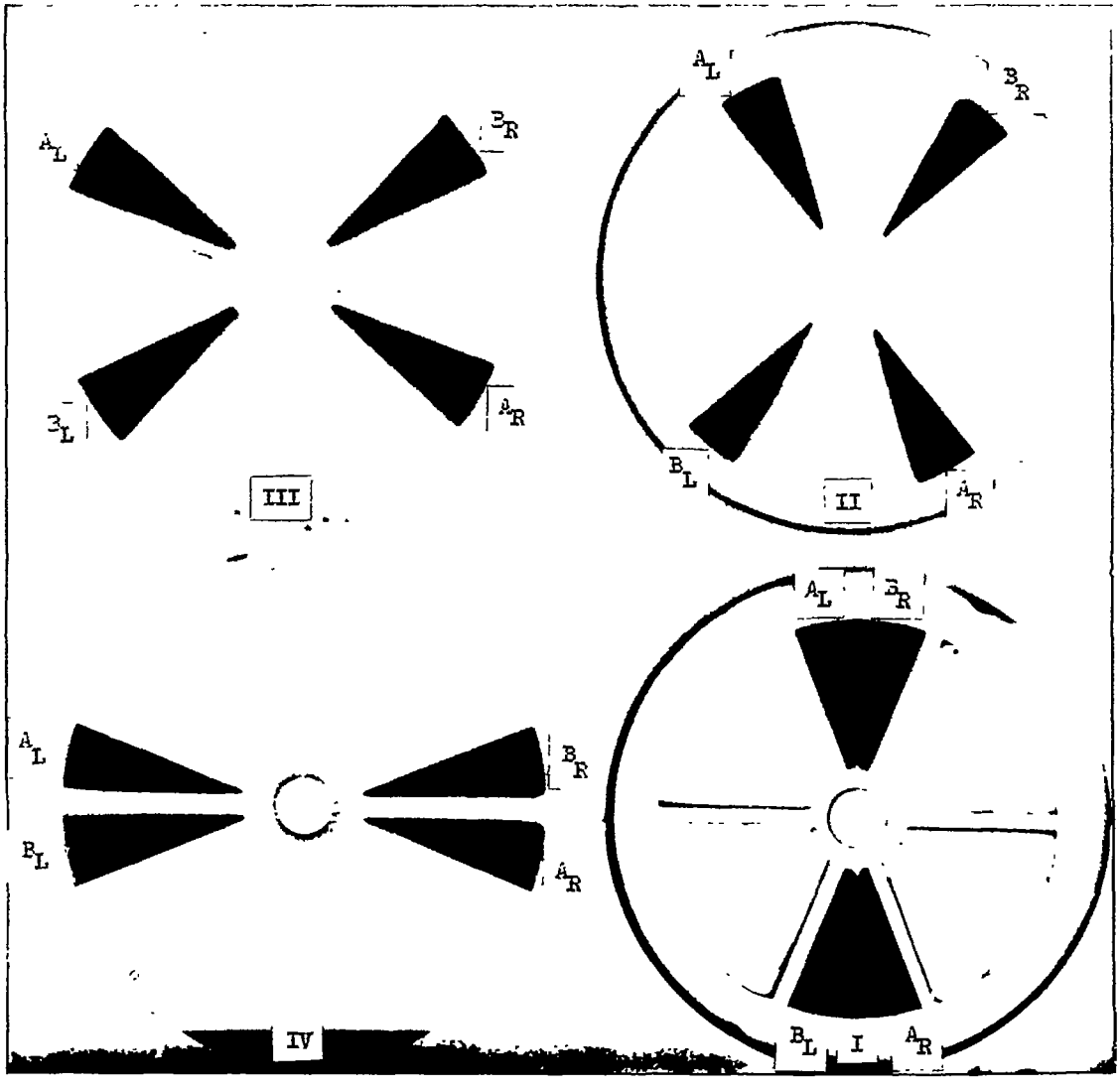

Figure 4. - Sample diffraction films for SAE X4130 steel.

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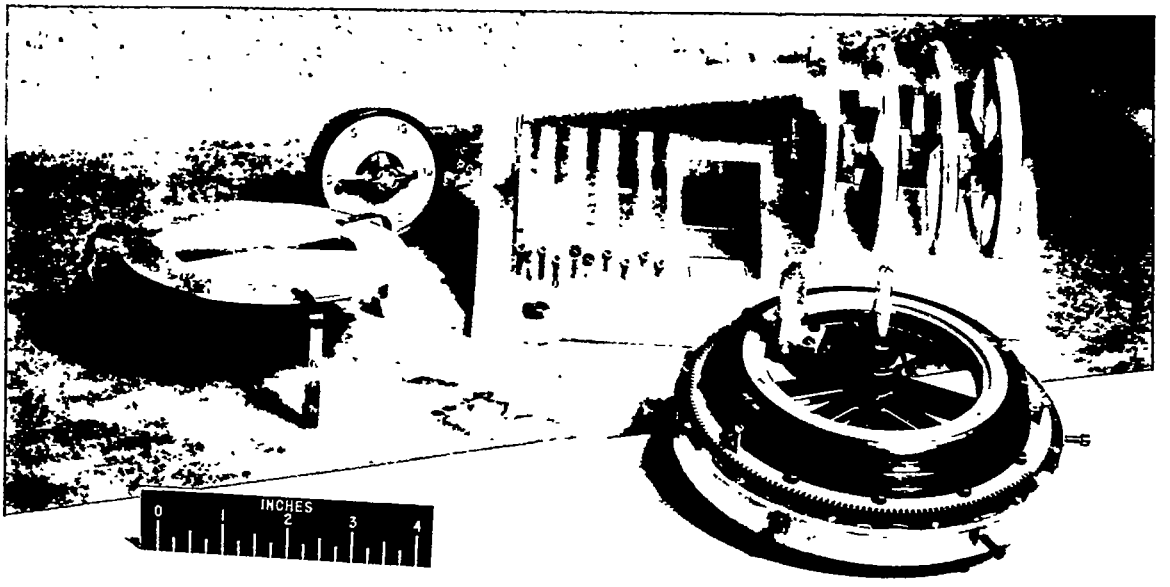

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Figure 5. - Loading and scribing operation with multiple-film camera.

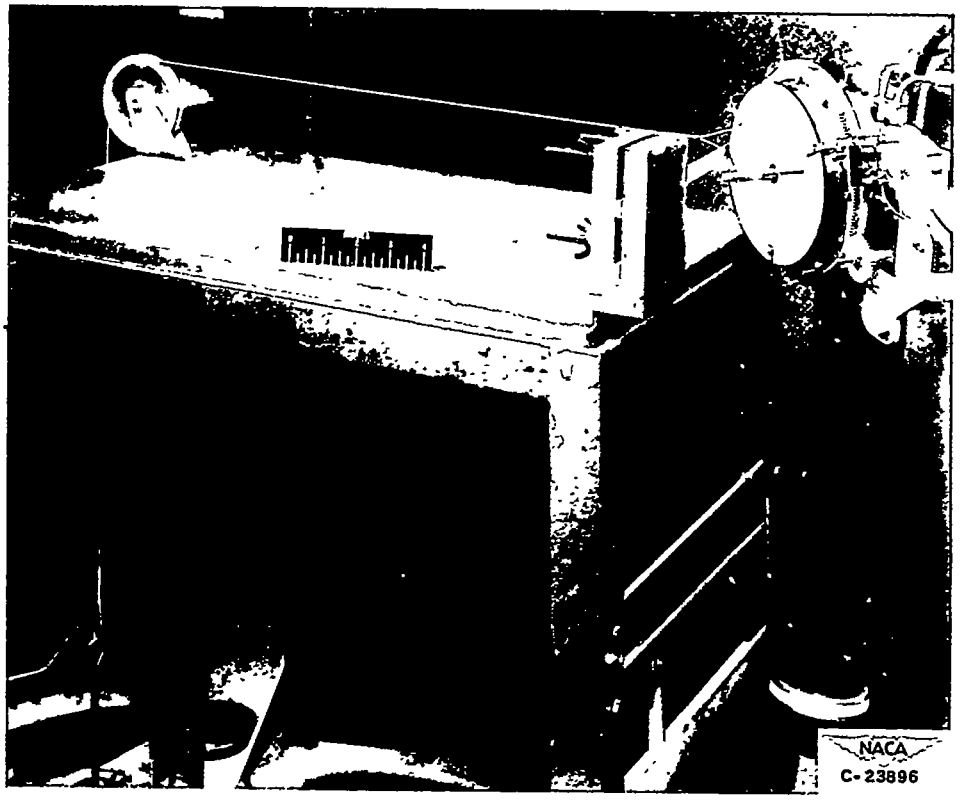

Figure 6. - Experimental setup for SAE X4130 steel check run.

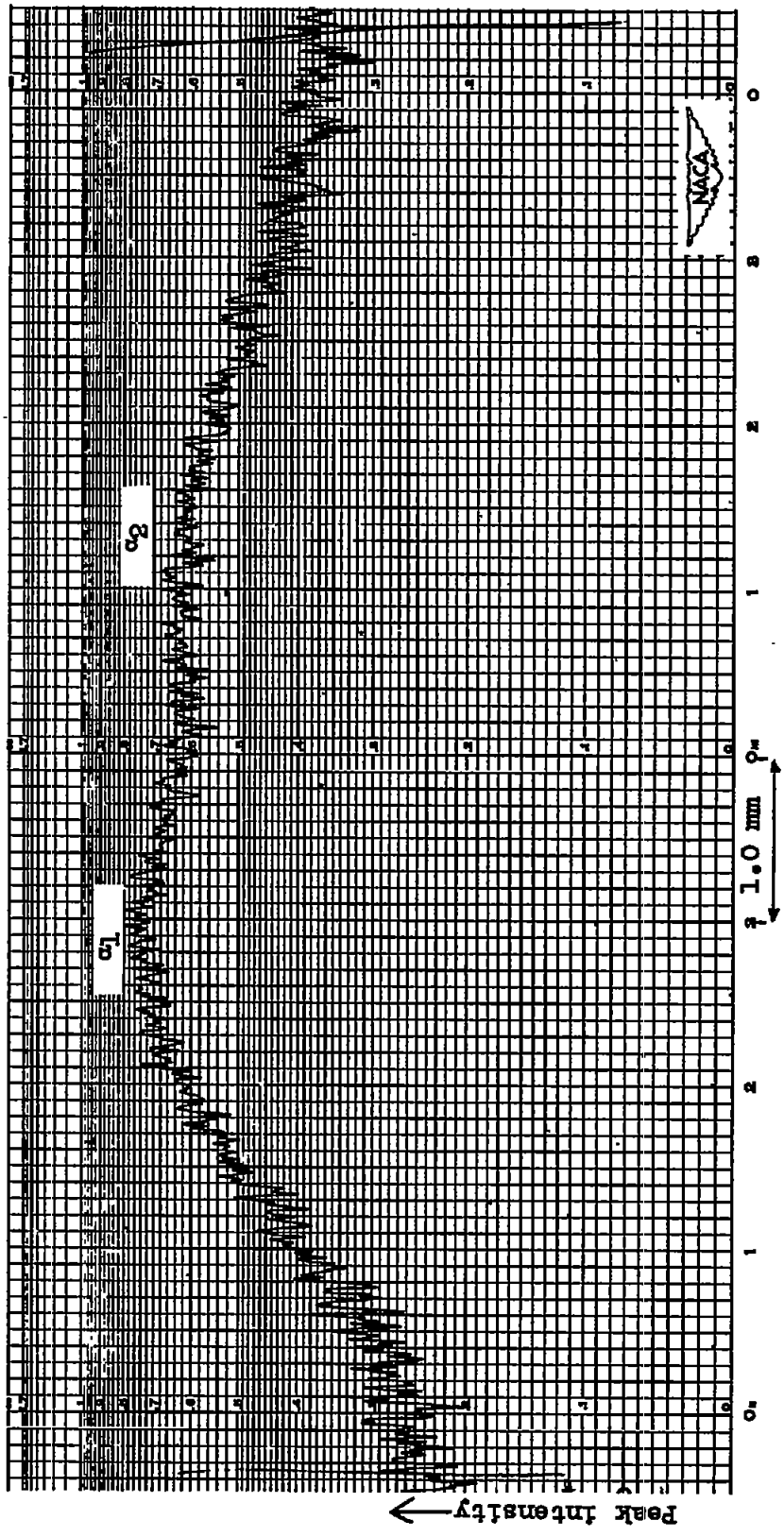

Figure 7. - Microphotometer trace of diffuse pattern of 310 planes of SAE X4130 steel.

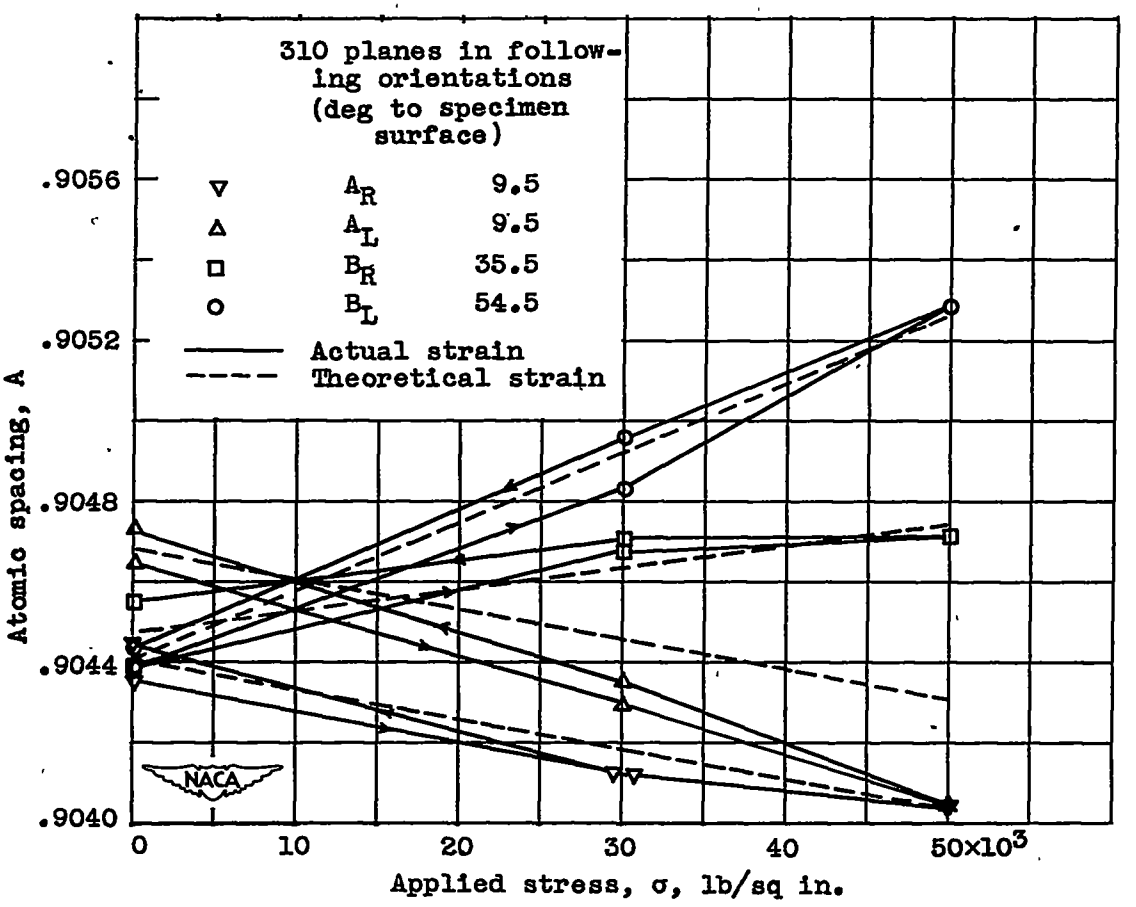

Figure 8. - Change in atomic spacing with applied stress for SAE X4130 steel.

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